भारतीय मानक
Indian Standard

IS 3025 (Part 27/Sec 1) : 2021

जल एवं अपशिष्ट जल के नमूने लेने तथा परीक्षण (भौतिक एवं रसायन) की पद्धतियां

भाग 27 साइनाइड

अनुभाग 1 टाइट्रीमेट्रिक, कलरमेट्रिक और आयन-सेलेक्टिव तरीके

(दूसरा पुनरीक्षण)

Methods of Sampling and Test (Physical and Chemical) for Water and Waste Water

Part 27 Cyanide

Section 1 Titrimetric, colorimetric and ion-selective methods

(Second Revision)

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Water Quality Sectional Committee, CHD 36

FOREWORD

This Indian Standard (Part 27/Sec 1) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Water Quality Sectional Committee had been approved by the Chemical Division Council.

This standard was first revised in 1986. The first revision had included colorimetric and ion selective electrode methods. In this revision, the following changes have been made:

a) Isolation procedure of cyanide from sample is separately prescribed followed by test methods for evaluation of cyanide content in sample;
b) Titrimetric method has been added;
c) Scope has been revised to explain the different methods of determination and their respective ranges; and
d) Precautions for handling cyanide solutions and method of disposal of cyanide waste included.

Many of the cyanides in soil and water come from industrial processes. The major sources of cyanides in water are discharges from some metal mining processes, organic chemical industries, iron and steel plants or manufacturers, and publicly owned wastewater treatment facilities. Other cyanide sources include vehicle exhaust, precious metals refining industries, burning of municipal waste, and use of cyanide-containing pesticides. Hydrogen cyanide, sodium cyanide, and potassium cyanide are the forms of cyanide most likely to be in the environment as a result of industrial activities. Cyanide compounds with free cyanide ions are acutely toxic to humans, animals and most other species. Presence of cyanide in water severely affects both aquatic life and humans coming in contact with the water. The lethal dose (LC 50 or LD 50) for gaseous hydrogen cyanide is 100-300 ppm and LD 50 for ingestion is 50-200 mg or 1-3 mg/kg body weight, whereas LD 50 for contact with unabraded skin is 100 mg (as HCN) per kg of body weight. Hence, it is necessary to determine the cyanide content of water and waste water.

This section of Part 27 covers the titrimetric, colorimetric and ion-selective methods for determination of total and free cyanides. Section 2 of Part 27 covers the flow injection analysis method whereas Section 3 covers continuous flow analysis method.

For the determination of cyanide, first various interfering substances like sulphides, oxidizing agents, nitrites and nitrates are removed. After removal of interfering substances, the metal cyanide is converted into hydrogen cyanide gas, which is distilled and absorbed in sodium hydroxide solution. The absorption liquid can be analyzed by titrimetric, colorimetric or cyanide ion selective electrode procedure. In view of the acute toxicity of cyanide compounds, it is recommended that all tests prescribed in this standard should be performed by qualified chemists and required safety measures should be ensured in the laboratory.

The composition of the Committee, responsible for the formulation of this standard is given at Annex A.

In reporting the results of a test or analysis in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTE WATER

PART 27 CYANIDE

Section 1 Titrimetric, colorimetric and ion-selective methods

(Second Revision)

1 SCOPE

This standard (Part 27/Sec 1) prescribes methods for the determination of total cyanide in water and waste waters. As there are so many interferences due to metals like iron, copper and nickel the preliminary acid distillation is a prerequisite and the distillate collected in alkaline scrubbing medium can be analyzed with 3 methods based on cyanide concentration. The cyanide ion exists in 3 forms that is, inorganic cyanide in water in CN^- ion form, undissociated hydrogen cyanide, or as anionic stable complexes with metal cations like iron, cadmium, zinc, mercury.

After removal of various interfering substances like sulphides, oxidizing agents, nitrites and nitrates, all the cyanides are converted into HCN gas in acidic medium and distilled and absorbed in sodium hydroxide solution. The absorption liquid of distillate can be used to estimate the cyanide by any of the following methods:

a) Titrimetric method (used if cyanide concentration is above 1 mg/l);
b) Colorimetric method (if cyanide concentrations are from 1 to 5 µg/l); and
c) Ion sensitive electrode method (if cyanide ranges from 0.05 to 10 mg/l).

Method detection limit : 0.02 mg/l.

2 DETERMINATION OF TOTAL CYANIDE AFTER DISTILLATION

2.1 Principle

Distillation of sample in the presence of sulphuric acid converts simple and complex cyanides into hydrocyanic acid. The hydrogen cyanide gas is absorbed in a solution of sodium hydroxide.

For example: $Fe(CN)_6^{-4} + 6H^+ \dashrightarrow 6HCN + Fe^{+2}$

$$HCN + NaOH \dashrightarrow NaCN + H_2O$$

2.2 The cyanides consists of inorganic cyanides, metal complexes, HCN and cyanides amenable for chlorination, and thiocyanates and interferences like oxidizing agents, fatty acids, nitrites, nitrates, aldehydes, sulphides etc. and needs preliminary distillation.

2.3 Interferences

2.3.1 Sulphides and nitrites interfere, and should be removed prior to distillation (*see* **2.7**).

2.3.2 Other interfering substances are removed by distillation.

2.4 Sampling Procedure and Storage

2.4.1 The samples should be collected in 2 litre polyethylene bottle and analyzed as soon as possible after collection.

2.4.2 Samples should be preserved by addition of sufficient sodium hydroxide to raise the *p*H to 11.0 or above and be stored in a cool place.

2.5 Apparatus

2.5.1 *Boiling Flask* — 1 litre, with inlet tube and provision for water-cooled condensers (*see* Fig. 1).

2.5.2 *Heating Mantle*

2.5.3 *Gas Absorber* — With gas dispersion tube equipped with medium-porosity fritted outlet.

2.5.4 *Ground Glass ST Joints* — TFE sleeved or with an appropriate lubricant for the boiling flask and condenser. Neoprene stopper and plastic threaded joints may also be used.

2.6 Reagents

2.6.1 *Sodium Hydroxide Solution* — Dissolve 50 g sodium hydroxide in 1 litre distilled water.

2.6.2 *Lead Carbonate* — Powdered.

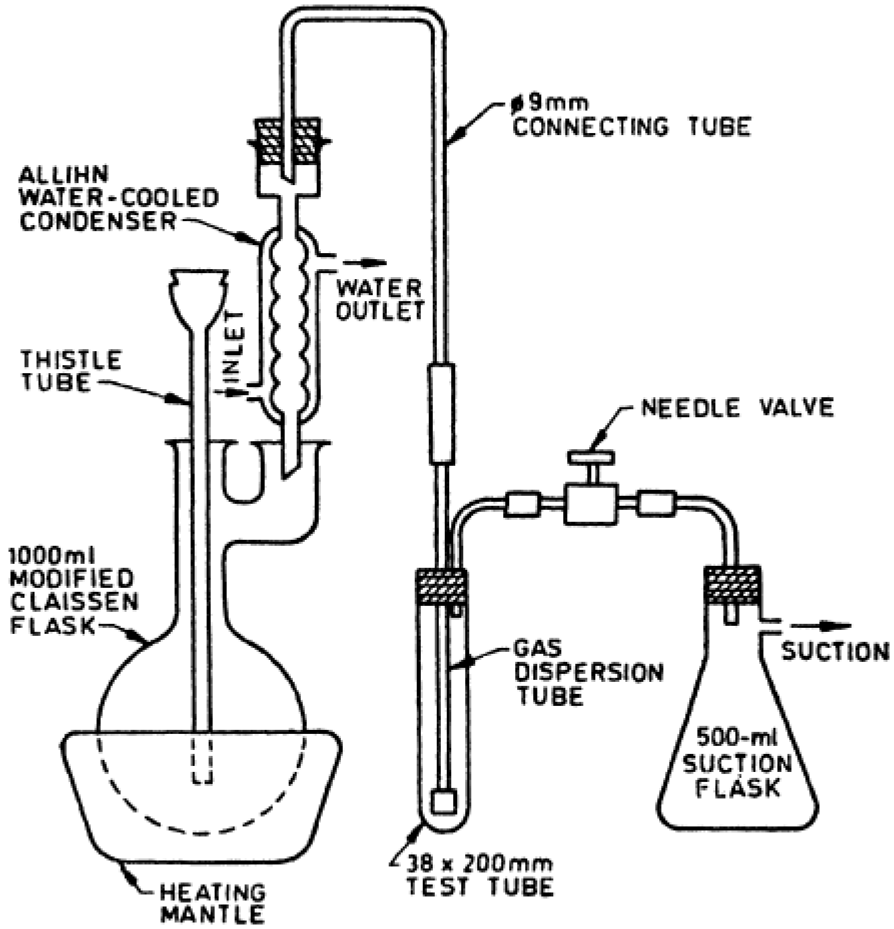

FIG. 1 CYANIDE DISTILLATION APPARATUS

2.6.3 *Sulphamic Acid* (NH_2SO_3H).

2.6.4 *Magnesium Chloride Solution* — Dissolve 51 g magnesium chloride ($MgCl_2.6H_2O$) in 100 ml distilled water.

2.6.5 Sulphuric Acid Concentrated.

2.6.6 *Acetic Acid* — Make by diluting 1 part of glacial acid with 4 parts of water.

2.7 Procedure

2.7.1 *Distillation*

2.7.1.1 Add 500 ml sample to the boiling flask. Add 10 ml of sodium hydroxide solution to gas scrubber and dilute, if necessary, with distilled water to obtain an adequate liquid depth in the absorber. Do not use more than 225 ml total volume of absorber solution. When sulphide generation from the distilling flask is anticipated, add 50 or more mg powdered lead carbonate to the absorber solution to precipitate sulphide. Connect the train, consisting of boiling flask air inlet, flask condenser, gas washer, suction flask trap and aspirator. Adjust suction so that approximately 1 air bubble per second enters the boiling flask. This air rate will carry hydrogen cyanide gas from flask to absorber and usually will prevent a reverse flow of hydrogen cyanide gas through the air inlet. If this air rate does not prevent sample backup in the delivery tube, increase air flow rate to 2 air bubbles per second. Observe air purge rate in the absorber where the liquid level should be raised not more than 6.5 to 10 mm. Maintain air flow through the reaction.

2.7.1.2 Add 2 g of sulphamic acid through the air inlet tube and wash down with distilled water.

2.7.1.3 Add 50 ml of concentrated sulphuric acid through the air inlet tube with distilled water and let air mix flask contents for 3 min. Add 20 ml of magnesium chloride reagent through air inlet and wash down with stream of water. A precipitate that may form redissolves on heating.

2.7.1.4 Heat with rapid boiling, but do not flood condenser inlet or permit vapours to rise more than halfway into condenser. Adequate refluxing is indicated by reflux rate of 40 to 50 drops/min from the condenser lip. Reflux for at least 1 h. Discontinue heating but continue air flow. Cool for 15 min and drain gas washer contents into separate container. Rinse connecting tube between condenser and gas washer with distilled water, and rinse water to drained liquid, and make up to 250 ml in a volumetric flask.

3 CYANIDE ANLYSIS BY USING TITRIMETRIC METHOD

3.1 Principle

The cyanide from alkaline distillate is titrated with standard silver nitrate to form a soluble silver cyanide complex AgCN. After all the cyanide ion complexes, the excess silver ion can be detected by using silver sensitive indicator that is, p-dimethylamino-benzalrhodanine. This indicator turns from a yellow color to salmon color. If cyanide is below 1 mg/l this method is not suitable.

3.2 Apparatus — Koch micro burette of 10 ml capacity.

3.3 Reagents

3.3.1 *Indicator Solution* — Dissolve 20 mg of p-dimethyl amino benzal rhodanine in 100 ml acetone.

3.3.2 *Silver Nitrate Standard Solution* — Dissolve 3.27 g of silver nitrate in 1 liter of distilled water and standardize with standard Sodium chloride (0.0141 N) solution using the potassium chromate indicator as specified in chloride estimation. Based on the titer value dilute 500 ml or equivalent amount to 1 000 ml so that a solution of 1 ml ≡ 1 mg CN^-.

3.3.3 *Sodium Hydroxide Solution* — Dissolve 1.6 g NaOH in 1 liter distilled water.

3.4 Procedure

Take 10 ml of cyanide distillate from the absorption flask and dilute to 100 ml by using NaOH solution. Add 0.5 ml indicator solution and titrate with silver nitrate till a first change of colour from canary yellow to a salmon hue occurs.

Similarly titrate a blank containing 10 ml of distilled water, diluted to 100 ml with NaOH solution with 0.5 ml indicator solution against silver nitrate till colour change appears.

3.5 Calculation

$$\text{mg CN}^-/\text{l} = (A - B) \times \frac{1000}{V_1} \times \frac{250}{V_2}$$

where

A = volume of $AgNO_3$ consumed by sample, in ml;
B = volume of $AgNO_3$ consumed by blank, in ml;
V_1 = volume of sample = 100 ml; and
V_2 = volume of cyanide distillate = 10 ml.

4 COLORIMETRIC METHOD (PYRIDINE-BARBITURIC ACID METHOD)

4.1 Principle

The cyanide ion of alkaline distillate after preliminary treatment is converted into cyanogen chloride (CNCl) by reaction with chloramine-T. The cyanogen chloride reacts with pyridine-barbituric acid reagent to form a red-blue color complex. The absorbance can be measured between 575-582 nm.

4.2 Apparatus — Spectrophotometer with a light path of at least 10 mm or longer to measure absorbance at 578 nm.

4.3 Reagents

4.3.1 *Chloramine-T Solution* — Dissolve 1.0 g of this reagent into 100 ml distilled water and store in refrigerator. The life of reagent is one week only.

4.3.2 *Stock Cyanide Solution* — Dissolve 2.51 g potassium cyanide in 1 litre water, standardize this solution with 0.019 2 N silver nitrate solution. The solution loses strength gradually and must be rechecked every week (1 ml of this solution ≡ 1 mg CN^-).

CAUTION — Potassium cyanide is highly toxic, take care to avoid ingestion, use gloves while preparing solution.

4.3.3 *Standard Ccyanide Solution* — Dilute 10 ml stock cyanide solution (4.3.2) to 1 litre with distilled water, mix and make a second dilution of 10 ml to 100 ml. One ml ≡ 1 µg CN^-.

NOTE — This solution must be prepared daily.

CAUTION — Toxic, take care to avoid ingestion. While taking aliquot of stock solution, use suction device coupled with pipette for drawing aliquot.

4.3.4 *Standard Silver Nitrate Solution* — Dissolve 3.27 g of silver nitrate in 1 litre of distilled water. Store in dark bottle. 1 ml of this solution ≡ 1.00 mg CN^-.

4.3.5 *Pyridine-Barbituric Acid Reagent* — 15 g of barbituric acid is added into 250 ml volumetric flask and dissolved with small amount of water. Add 75 ml of Pyridine and mix thoroughly. Add 15 ml of concentrated hydrochloric acid and mix thoroughly and cool it to room temperature. Dilute with distilled water and store in amber colour bottle in refrigerator. This solution is stable and lasts for 6 months. If precipitate develops, discard immediately.

4.3.6 *Sodium Hydroxide Solution* (0.2 N) — Dissolve 8.0 g sodium hydroxide in 1 l distilled water.

4.3.7 *Acetate Buffer* — Dissolve 410 g sodium acetate trih ydrate in 500 ml water and add glacial acetic acid to adjust to *p*H 4.5 and make it up to 1 liter water.

4.4 Procedure

4.4.1 *Preparation of Standard Curve*

Prepare a series of standards containing 1 to 10 µg CN^- into 50 ml volumetric flasks (0.02-0.2 µg CN^-/ml). Dilute to 40 ml with NaOH solution. Add 1 ml of acetate buffer and 2 ml chloramine-T and mix it by closing stopper for 2-3 minutes. Add 5 ml pyridine-barbituric acid reagent and makeup to 50 ml with distilled water and let stand for 8 minutes to develop colour. Measure absorbance against distilled water at 578 nm.

Use a separate flask with 40 ml NaOH as blank solution. Add 1 ml of acetate buffer and 2 ml chloramine-T and mix it by closing stopper for 2-3 min. Add 5 ml pyridine – barbituric acid reagent and makeup to 50 ml with distilled water. Measure the absorbance of blank at 578 nm.

The standard curve is plotted by taking the net absorbance (difference between absorbance of cyanide standards and absorbance of blank) on the abscissa and concentrations of cyanide on the ordinate.

4.4.2 *Measurement of Sample*

Take 10 ml of the cyanide distillate into 50 ml volumetric flasks and make up the volume to 40 ml with NaOH solution. Thereafter add reagents as given in 4.4.1 and measure absorbance against distilled water at 578 nm.

4.5 Calculation

Calculate the cyanide concentration as follows from slope and intercept of standard curve drawn

$$CN^- \text{ in mg/l} = (\text{Slope} \times A_1 + \text{intercept}) \times \frac{50}{X} \times \frac{250}{Y}.$$

where

X = volume of original sample = 500 ml,

Y = volume of sample taken for colorimetric measurement = 10 ml, and

A_1 = absorbance of sample solution.

5 SELECTIVE ION ELECTRODE METHOD

5.1 Principle

Cyanide in the alkaline distillate from the preliminary treatment, as given in **2.7.1**, can be determined potentiometrically by using a cyanide in selective electrode in combination with a double junction reference electrode and a *p*H meter having an expanded millivolt scale, or specific ion meter.

5.2 Apparatus

5.2.1 *Expanded-scale* pH *Meter or Specific Ion Meter*

5.2.2 *Cyanide-ion-selective Electrode*

5.2.3 *Reference Electrode, Double-junction*

5.2.4 *Magnetic Mixer with TFE-coated Stirring Bar*

5.3 Reagents

5.3.1 *Stock standard Cyanide Solution — see* **4.3.2.**

5.3.2 *Sodium Hydroxide Diluent* — Dissolve 1.6 g sodium hydroxide in water and dilute to 1 l.

5.3.3 *Intermediate Standard Cyanide Solution* — Dilute a calculated volume (approximately 100 ml) of stock potassium cyanide solution, based on the determined concentration, to 1 000 ml with sodium hydroxide diluent. Mix thoroughly 1 ml ≡ 100 µg $CN^–$.

5.3.4 *Dilute Standard Cyanide Solution* — Dilute 100 ml intermediate cyanide standard solution to 1 000 ml with sodium hydroxide diluent, 1.00 ml ≡ 10.0 µg $CN^–$. Prepare daily and keep in a dark, glass-stoppered bottle.

5.3.5 *Potassium Nitrate Solution* — Dissolve 100 g potassium nitrate in water and dilute to 1 litre. Adjust to *p*H 12 with potassium hydroxide. This is the outer filling solution for the double junction reference electrode.

5.4 Procedure

5.4.1 *Calibration*

Use the dilute and intermediate standard cyanide solutions and sodium hydroxide diluents to prepare a series of three standards, 0.1, 1.0 and 10.0 mg $CN^–$/l. Transfer approximately 100 ml of each of these standard solutions into a 250 ml beaker pre-rinsed with a small portion of standard being tested. Immerse $CN^–$ and double-junction reference electrodes. Mix well on a magnetic stirrer at 27 °C, maintaining as closely as possible the same stirring rate for all solutions.

Always progress from the lowest to the highest concentration of standard otherwise equilibrium is reached only slowly. The electrode membrane dissolves in solutions of high cyanide concentration; do not use with a concentration above 10 mg/l. Hence, it is recommended to remove electrodes from test solution immediately after measurement and soak in water.

After equilibrium is reached (at least 5 min and not more than 10 min), record potential (millivolt) readings and plot $CN^–$ concentrations *versus* readings on semi-logarithmic graph paper. A straight line with a slope approximately 59 mV per decade indicates that the instrument and electrodes are operating properly. Record slope of line obtained (millivolts/decade of concentration). The slope may vary somewhat from the theoretical value of 59.2 mV per decade because of manufacturing variation and reference electrode (liquid-junction) potentials. The slope should be a straight line and is the basis for calculating sample concentration.

5.4.2 *Measurement of Sample*

Place 100 ml of absorption liquid obtained in **2.7.1** into a 250 ml beaker. When measuring low cyanide concentrations, first rinse beaker and electrodes with a small volume of sample. Immerse cyanide and double-junction reference electrodes and mix on a magnetic stirrer at the same stirring rate used for calibration. After equilibrium is reached (at least 5 min and not more than 10 min), record values indicated on ion meter or found from graph prepared above. Calculate concentration as given below.

5.5 Calculation

$$\text{Cyanide, mg/l} = \text{mg CN}^{-}\text{/l from meter} \times \frac{1000}{X} \times \frac{250}{Y}$$

where

X = volume of original sample = 500 ml, and

Y = volume of sample taken for measurement = 100 ml.

6 SAFETY PRECAUTIONS FOR HANDLING CYANIDE SOLUTIONS

6.1 Cyanide compounds are highly poisonous and they can prevent the utilization of oxygen by body tissues. If cyanide compounds come in contact with the skin or eyes, they shall be washed off immediately as they can be absorbed through the skin.

6.2 Protective clothing, such as a laboratory coat, mask, and latex gloves must be worn when handling NaCN or KCN tablets and cyanide solutions.

6.3 A fume hood must be used to prepare all cyanide solutions and to house the distillation apparatus (equipped with alkaline scrubbing facility) used for cyanide testing.

6.4 All used cyanide solutions must be stored in dark-colored bottles with pellets of sodium hydroxide added. The pre-treatment of cyanide solutions with NaOH pellets must be done prior to their disposal.

6.5 Prior to disposal, cyanide solutions shall be decomposed with sodium hypochlorite solution (concentration approximately equal to 17.3 times the cyanide concentration and $p\text{H} \sim 12$)[1] in alkaline medium. Disposal shall be done in a sink with copious amounts of tap water.

6.6 Use of alkaline solution is essential for decontamination of cyanide waste. In case of accidental spills or the splattering of chemicals or samples, contaminated areas should be promptly rinsed with large amounts of water and alkaline water. Medical treatment/advice shall be sought immediately.

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ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Water Quality Sectional Committee, CHD 36

Organization	*Representative(s)*
Chief Scientist, EPTRI, Hyderabad	Shri N. Raveendhar (***Chairman***)
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